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DEVELOPMENT OF ULTRA-THIN FILM PRESERVATIVE COMPOUNDS

Prepared under Navy, Bureau of Naval Weapons
Contract NOw 61-0855c

FINAL REPORT

6 June 1961 through 5 July 1962

FOSTER D. SNELL, INC.
29 W. 15th Street, N. Y.

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ABSTRACT

Candidate materials were tested in a controlled cyclic condensation humidity cabinet. Several of these materials meet the corrosion preventive requirements of an ultra-thin film preservative compound. Three of these were tested with the Falex Lubricant Testing Machine for effect on wear properties of a typical lubricant.

Alox 2028 (a mixture of organic acids, oxyacids and esters derived from oxidation of petroleum hydrocarbons) appears to be the most satisfactory material tested.

The controlled cyclic condensation humidity cabinet used as the corrosion test cabinet for this project was developed by D Minuti and J. E. Carroll of Aeronautical Materials Laboratory, Naval Air Material Center, Philadelphia, Pennsylvania. It was modified by this laboratory to permit greater numbers of samples to be tested at one time.

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I. SUMMARY

This is the final report to the Bureau of Naval Weapons under Contract NOw 61-0855c on the development of an ultra-thin film preservative compound.

The most promising composition developed under the terms of the contract consisted of a 50% solution of Alox 2028 in a toluene-butanol blend. Alox 2028 is a mixture of carboxylic acids, oxyacids and esters obtained from oxidation of petroleum hydrocarbon fractions. This has been found to provide complete protection to polished, ferrous metal surfaces for up to 48 hours, under controlled cyclic condensation conditions in film thickness of 0.0003 inches. In addition, it has been found to have no adverse effect on wear properties of a petroleum-type lubricant.

Other materials found to give complete protection for the 48 hour period in the controlled cyclic condensation humidity cabinet were Acryloid B-72, Emralon 320, Gelva C5-V16, Gulf No-Rust D, and zinc naphtenate. In addition, about one-third of the materials tested under this contract exhibited significant rust-preventive properties based on results obtained with the test cabinet (less than 10% surface rusted with protective films of less than 0.0005 inches).

Commercial products conforming to grade 3 materials of MIL-C-16173B were tested in the corrosion test cabinet and were used as a basis for comparison with other materials.

Three-hour wear tests using the Falex Lubricant Tester were performed in order to test the effects of some of those materials on a petroleum-type lubricant. There was no apparent change in wear due to coating of test pins and blocks with materials envisaged as possible preservative compounds.

II. INTRODUCTION

The purpose of this project was to develop formulations which when applied to critical metal surfaces would deposit an ultra-thin film (less than 0.0005 inches) as a non-tacky, preservative coating. It was desired that a satisfactory material for this purpose should have the following characteristics:

(1) The preservative coating shall be ultra-thin (less than 0.0005 inches thick), transparent and non-tacky.

(2) It shall afford a high degree of protection to critical metal surfaces.

(3) It shall not require removal prior to placement of the treated part in service.

(4) The preservative film shall not constitute a safety hazard in contact with oxygen, nor shall it support combustion in the absence of an applied flame.

(5) It shall not interfere with normal or dry-film lubrication.

(6) The preservative shall be applicable with solvents that are quick-drying and non-toxic. The compound shall be homogeneous, free from grit, abrasives, or other impurities and shall not be injurious in any way to personnel handling it if reasonable procedure and safety precautions are employed.

At the conclusion of this project, it appears that there are several materials which might satisfactorily answer these requirements. These materials are:

Alox 2028	(a mixture of carboxylic acids, oxyacids, and esters from oxidation of petroleum hydrocarbons)
Acryloid B-72	(acrylic ester polymer)
Emralon 320	(tetra fluoroethylene surface coating)
Gelva C5-V16	(alkali-soluble vinyl acetate polymer)
Zinc naphthenate	

In addition, it seems quite likely that many more materials could be found from further testing which would answer the above requirements.

III. RECOMMENDATIONS

The recommended formula given below is considered the best ultra-thin film preservative compound tested thus far. It is quite possible that further formulation work and testing would reveal other materials equally capable of satisfying the aims of this project. The present compound does not completely meet the requirement that the film be non-tacky since Alox 2028 is a semi-solid material. If non-tackiness is considered an absolutely essential property, then a solution of zinc naphthenate can be considered an alternative recommendation. (Lack of lubricity testing on this material is the only reservation made concerning its feasibility).

Composition and Raw Material Costs of:

Formula #12	Parts by Weight	Cost per pound	Cost per 100 pounds of Compound
Alox 2028	50.0	34¢/lb	17.000
Toluene	33.0	4¢/lb	1.320
n-Butanol	17.0	19-1/2¢/lb	3.315
			21.635

Method of Manufacture

The manufacture of Preservative Compound, Formula #12, can be accomplished in any suitably sized clean container with appropriate mixing equipment. Since Alox 2028 is semi-solid and dissolves completely and readily in the solvent mixture, there is no need for restrictions in the order of addition of materials.

Results of Tests with Alox 2028

1. Corrosion Tests

Corrosion tests were conducted in the controlled cyclic condensation humidity cabinet; details of this test are described in another section of this report.

Test	Metal Finish (microns)	Film Thickness (mils)	Per Cent Surface Rusted
1	12	0. 2	1
2	4	0. 1	4
*2	4	0. 1	5
3	4	0. 4	0
4	1	0. 3	0
5	1	0. 3	0

*-film of Alox 2028 was applied over a previously applied film of titanium dioxide.

2. 3-Hour Wear Test

Measurement of wear of test pins and blocks was made using the Falex Lubricant Tester which was operated at 120 psi and 175°F. Test pins were of SAE 3135 steel of Rockwell B-90 hardness while "V" blocks were "open hearth" steel corresponding to AISI C-1137 grade steel of Rockwell C 20 hardness. A detailed description of the test is presented in another section of this report.

Depth of Wear (inches)

Alox 2028 coated pin	Uncoated pin	Change
0. 0001668	0. 0001668	0. 0000

Sources of Raw Materials

Alox 2028	Alox Corporation P. O. Box 556 Niagara Falls, N. Y.
Toluene	Amoco Chemical Corp. 130 E. Randolph Drive Chicago 1, Illinois

Sources of Raw Materials (con't)

n-Butanol	Union Carbide Chemicals Co. Division of Union Carbide Corp. 270 Park Avenue New York 17, N. Y.

IV. CORROSION TESTS

A. Controlled Cyclic Condensation Humidity Cabinet

Basically, the corrosion test cabinet is an insulated 28" x 28" x 28" chamber with thermostatically controlled heaters. Controlled humidity is achieved by drawing metered air from an outside thermostatically controlled water chamber. The humidified air flows through a heat exchanger which serves to pre-condition the air prior to entering the humidity cabinet. The air enters at the bottom and is diverted into upper and lower manifolds which diffuse the air throughout the cabinet. Test specimens are mounted over water-tight specimen holders containing a water bath. The temperature of the water, which intimately contacts the underside of the test specimen, is controlled to effect cyclic evaporation and condensation conditions on the test surface. Condensation is induced on the test specimen by passing a refrigerant, supplied by an external compressor unit, through a coil contained in the water bath of the specimen holder. Evaporation is induced by permitting the elevated ambient temperature of the test cabinet to heat the water bath above the dew point. The condensation-evaporation cycle is controlled by a programming unit. This cabinet can be operated over a range of ambient conditions from 70°F to 180°F and at 25% to nearly 100% relative humidity.

Among the advantages offered by this corrosion test cabinet are:

1. Conditions within the cabinet are unaffected by ambient conditions existing in the laboratory.

2. There are no irregularities caused by the washing action of condensate which induces rust streaks.

3. Conditions of test, although accelerated, more closely simulate the diurnal cycle of condensation and evaporation.

4. Results such as intensity and corrosion pattern are typical of field situations.

B. Preparation of Test Specimens

Steel coupons (2" x 4" x 1/8"), of SAE 1010 steel were used as test specimens. Surfaces were prepared as follows:

1. Irregularities, burrs, etc. were ground away from panel edges and corners using 150-grit Aluminum oxide wet-or-dry grinding belt running at 1800 rpm. During the grinding process, the belt was kept wet with kerosene.

2. Panels were rinsed in naphtha and the panel faces were ground using the same equipment and materials as in step 1.

3. After rinsing in naphtha, panel faces were again ground with the belt grinder using a 320-grit Aluminum oxide grinding belt.

3a. After rinsing clean, panels were polished with a metallurgical disc polisher using 320 mesh aluminum oxide powder wetted with kerosene. A canvas cloth was fitted over the disc polisher to contain the polishing grit and the disc was rotated at 230 rpm. The disc was rinsed frequently with kerosene and fresh polishing mixture added to the cloth during the polishing operation.

3b. After rinsing clear, panels were polished again as in (3a) using 1 micron aluminum oxide powder and microcloth polishing cloth.

4. Panels were washed with 1% solution of Triton X100 in kerosene.

5. Panels were degreased in trichloroethylene vapor-degreaser.

6. Panels were rinsed with hot methanol, then in naphtha and allowed to dry.

7. Test coupons were dip-coated from respective solutions of test materials using a Fischer-Payne dip-coater operated at a withdrawal rate of 1/16 inch per second. Panels were allowed to dry overnight before being placed in the test cabinet.

Steps 3a. and 3b. were omitted from the initial tests. For later tests, a commercial polishing firm finished the panels.

C. Operation of Corrosion Test Cabinet

Operation of the controlled cyclic condensation humidity cabinet for the corrosion tests conducted for this project was accomplished in the following manner:

After all test specimens had been coated with respective test materials and had been dried overnight, panels were placed in their designated spaces over filled water jackets. Care was taken to insure that there would be intimate contact between the water and the undersides of the test panels.

Before the panels were mounted in place all settings on the various controls and valves were made and heaters and air flow valves opened. When the panels were securely mounted the test period began almost immediately. The settings that were made were as follows:

Rate of Air Flow-	2 cu. ft. /minute
Moisturizing chamber-water temperature-	41°C ± 2°C
Heat exchanger-oil bath temperature-	70°C ± 5°C
Test cabinet - air temperature-	50°C ± 2°C
(dew point)	40°C
(relative humidity)	52%
Compressor operation cycle	On - 45 min.
(controlled by programming clock)	Off - 135 min.
Total Cycle-	180 min.

Refrigeration return valves were adjusted for each water jacket to control the rate of cooling. The following test conditions prevailed during operation of the equipment:

The surface temperatures of the test panels varied between 15°C at the low point (this was erroneously reported as 10°C in quarterly report #3) about 15 minutes after the refrigeration cycle shut off to 50°C (the cabinet air temperature) for the 15 minute period immediately preceding the next refrigeration "on" cycle and the first 5 minutes of the "on" cycle. This temperature cycle resulted in a condensation-evaporation period of 105 minutes when condensed moisture was present on the panels and 75 minutes when the test surface was dry.

D. Discussion of Results

Analysis of surface rusting data from these corrosion tests indicate the standard deviations of results to be about 3-1/2%. Since most of the testing period was during non-observable times, it is not possible to determine variations with respect to times of initial failure. During these tests, the average surface rusting observed on all panels tested was 25%.

An insufficient number of materials were retested, to permit definitive conclusions to be drawn regarding the repeatibility of results obtained with the controlled cyclic condensation humidity cabinet. However, when allowances are made for the different degrees of surface finish and differences in film thickness, the results obtained from retesting of several materials indicate that repeatibility of results is quite high with this equipment.

Of the various classes of materials tested, it appears that metal salts of organic acids offer the most promising degree of corrosion preventive properties.

Several commercial proprietary preservative compounds which, conform to grade 3 of Military Specification MIL-C-16173B were applied by dipping in solutions as received and in solutions diluted to

50% of original with toluol. The film thicknesses obtained from original samples ranged from 0.4 mils to 1.0 mils. However, thicknesses resulting from diluted solutions were less than 0.1 mil in every case. Even the thinnest films were too dark to permit observation of corrosion and had to be washed off test panels before any observations could be be made. It is interesting to note that one of these materials, Clarco #4000, failed to give significant protection to the metal surface even when applied in a 0.4 mil thick film. Only Gulf No-Rust D gave complete protection for the period of the test when applied from the thinned down solution. The other materials, Nokorode 733A and Cosmoline 1091 gave excellent protection as 0.8 mil films, and appear to retain much of their effectiveness in thinner films.

Table III lists the materials tested during this contract period which give promise of furnishing equal corrosion protection in ultrathin films as do those products qualified as grade 3 materials under specification MIL-C-16173B. Two of these, Emralon 320 and Zinc naphthenate, did not permit any surface rusting during the 48-hour test period even though film thicknesses were less than 0.1 mil.

The results for several of the materials were surprisingly good considering the nature of the film obtained. Ammonium stearate, calcium oleate, Deriphat 170C, and Hyfac 430 yielded irregularly semi-opaque films which indicates they were less than complete films. Yet each of these materials kept surface rusting below 10%.

The large proportion (approximately one-third of the materials tested) of materials which show significant corrosion-preventive ability in thin films is a hopeful portent that a large number of materials might be found to satisfy the aims of this project. The fact that at least one material of each class tested might be deemed as being significantly protective against corrosion reinforces this opinion.

V. EFFECT OF SOME ULTRA-THIN FILMS ON METAL WEAR

Films of Alox 2028, Acryloid B-72 and Gelva C5-V16, three materials which appeared to possess significant corrosion preventive properties, were applied to test pins and blocks of the Falex Lubricant Testing Machine. Three-hour wear tests were run with the pins and blocks immersed in an oil bath maintained at 175°F. The results obtained were compared with the results obtained with uncoated test pieces.

The Falex Lubricant Testing Machine is essentially a device in which a pin is rotated at 290 r.p.m. between two "V"-shaped bearing blocks which are inserted in two lever arms. The lever arms constitute a load applying mechanism. Load and torque gauges on the equipment facilitate proper control and recording of the required measurements. The load on the lever arms is applied by turning an attached ratchet wheel which serves as an accessory wear measurement device.

Using the ratchet wheel as a micrometer, the amount of wear on test pins was determined. This measurement is obtained by scribing the ratchet wheel at the beginning of the test while the test pieces are under a constant load and recording the number of teeth that are taken up to maintain this load over a three-hour period. Each tooth of the ratchet wheel taken up is equivalent to 0.0000556 inches of wear. Temperature, torque and tooth wear were recorded every 15 minutes. A constant load of 120 pounds was maintained for the three-hour test period.

The oil used during this test was a mid-continent distilled-solvent refined oil with a Viscosity Index of 90. In addition, this oil contained an oxidation-corrosion inhibitor and a V. I. improver.

The values obtained from these tests indicate that ultra-thin films of these materials can be expected to have little if any effect on normal lubrication properties. In two cases the wear obtained (0.00001668 inches for Acryloid B-72 and Alox 2028 coated pins) was identical to that obtained for uncoated pins. On the pin coated with Gelva C5-V16 the wear (0.0002224 inches) was slightly higher. Scar widths and microscopic examination of the scars on the "V" block test pieces indicate Gelva C5-V16 coated blocks to be the least affected (contrasting somewhat with the readings obtained from ratchet wheel). Alox 2028 coated blocks appeared next best while Acryloid B-72 coated and uncoated blocks appeared to have the most irregular scarring.

VI. MEASUREMENT OF FILM THICKNESS

Film thickness measurements during the course of this project were made with an Elcometer Magnetic thickness gauge in the case of dry films and a "wet-film" thickness gauge for others. Other methods which could provide more accurate measurements were studied, but replies from manufacturers of such instruments left the impression that little could be gained from a change to other methods.

One manufacturer, for example, suggested a beta radiation gauge utilizing the back scattering principle. However, besides being prohibitively expensive, this instrument had the limitation that the surface geometry of the base metal remains absolutely constant. In general, a requirement of almost all instruments was that the thickness of the base metal be controlled to a higher order of accuracy than that required of the thickness measurement of the film. This provision, obviously, militates against the use of many of these instruments for laboratory scale work.

VII. CONCLUSIONS

Effective preservation of critical metal surfaces can be accomplished with ultra-thin film preservative compounds.

The controlled cyclic condensation humidity cabinet (developed by Minuti and Carroll and modified by this laboratory) will make a useful research tool in corrosion studies.

Respectfully submitted,

FOSTER D. SNELL, INC.

William Miglas
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Research Chemist

Bernard Berkeley
Bernard Berkeley, Director
Product Development Dept.

WM:BB/aw
oz.
July 20, 1962

RESULTS OF TESTS

TABLE I

FORMULATIONS

Formula Number	Composition (active ingredient underlined)	Per Cent by Weight
#1	Acryloid B-72 (40%)	25.0
	Methyl ethyl ketone	25.0
	Toluene	30.0
	n-Butanol	20.0
#2	a. Acryloid B-72 (40%)	24.5
	b. Neutral barium petronate	0.2
	Methyl ethyl ketone	25.1
	Toluene	30.2
	n-Butanol	20.0
#3	Acryloid B-72 (40%)	75.0
	Toluene	25.0
#4	Acryloid B-72 (40%)	62.5
	Toluene	37.5
#5	Acryloid B-72 (40%)	50.0
	Toluene	50.0
#6	Acryloid B-72 (40%)	37.5
	Toluene	62.5
#7	Alamac 26D	4.9
	Water	92.7
	n-Butanol	2.4
#8	Alamine 26D	49.0
	Ethyl acetate	33.0
	Acetone	15.0
	Toluene	3.0

Formula Number	Composition (active ingredient underlined)	Per Cent by Weight
#9	Alkaterge T	10.0
	Toluene	25.0
	Trichloroethylene	20.0
	Methyl chloride	25.0
	n-Butanol	20.0
#10	Alox 2018	47.4
	Toluene	47.4
	n-Butanol	5.2
#11	Alox 2028	10.0
	Toluene	25.0
	Trichloroethylene	20.0
	Methyl chloride	25.0
	n-Butanol	20.0
#12	Alox 2028	50.0
	Toluene	33.0
	n-Butanol	17.0
#13	Ammonium petronate	40.0
	Toluene	40.0
	n-Butanol	20.0
#14	Ammonium stearate (25%)	52.4
	Water	42.8
	n-Butanol	4.8
#15	Atpet 200	10.0
	Toluene	25.0
	Trichloroethylene	20.0
	Methyl chloride	25.0
	n-Butanol	20.0
#16	Calcium Oleate	31.6
	Toluene	52.6
	n-Butanol	15.8

Formula Number	Composition (active ingredient underlined)	Per Cent by Weight
#17	Clarco #4000	100.0
#18	Clarco #4000	50.0
	Toluene	50.0
#19	Corrosion Inhibitor	57.1
	Toluene	42.9
#20	Cosmoline 1091	100.0
#21	Cosmoline 1091	50.0
	Toluene	50.0
#22	Dilauryl thiodipropionate	26.3
	Toluene	57.9
	n-Butanol	15.8
#23	Deriphat 170 C	78.3
	Toluene	6.0
	n-Butanol	15.7
#24	Dodecyl benzene sulfonic acid	50.0
	Toluene	33.0
	n-Butanol	17.0
#25	Duomeen TDO	10.0
	Toluene	25.0
	Trichloroethylene	20.0
	Methyl chloride	25.0
	n-Butanol	20.0
#26	Emersol 150	10.0
	Methyl ethyl ketone	30.0
	Toluene	40.0
	n-Butanol	20.0

Formula Number	Composition (active ingredient underlined)	Per Cent by Weight
#27	Emralon 320	100.0
#28	Emtall 662	78.9
	Toluene	15.8
	n-Butanol	5.3
#29	Epon 1007	10.0
	Methyl ethyl ketone	27.0
	Toluene	45.0
	Ethyl acetate	18.0
#30	Epoxol 9-5	50.0
	Methyl ethyl ketone	20.0
	Toluene	20.0
	n-Butanol	10.0
#31	Epoxol 9-5	80.6
	Toluene	13.3
	n-Butanol	6.1
#32	Gelva C5-V16	25.0
	Ethyl acetate	50.0
	Toluene	5.0
	Methanol	7.0
	n-Butanol	13.0
#33	Glyceryl monoricinoleate	79.9
	Toluene	15.1
	n-Butanol	5.0
#34	Gulf No-Rust D	100.0
#35	Gulf No-Rust D	50.0
	Toluene	50.0

Formula Number	Composition (active ingredient underlined)	Per Cent by Weight
#36	Hyfac 430	28.6
	Butyl cellosolve	17.1
	Methyl ethyl ketone	8.6
	Ethyl acetate	11.4
	Toluene	20.0
	Sovasol #5	14.3
#37	Metholene 2218	50.0
	Toluene	50.0
#38	Methyl p-toluene sulfonate	94.0
	Toluene	5.0
	n-Butanol	1.0
#39	Myverol type 18-98	10.0
	Methyl ethyl ketone	30.0
	Toluene	40.0
	n-Butanol	20.0
#40	Naphthenic acids	84.6
	Toluene	10.3
	n-Butanol	5.1
#41	Neutral Barium Petronate	10.0
	Methyl ethyl ketone	30.0
	Toluene	40.0
	n-Butanol	20.0
#42	Nokorode 733A	100.0
#43	Nokorode 733A	50.0
	Toluene	50.0
#44	Nopcochex RD	78.8
	Toluene	21.2

Formula Number	Composition (active ingredient underlined)	Per Cent by Weight
#45	No-Ox-Id 493	100.0
#46	Nacconol 60s	75.0
	Ethyl alcohol	25.0
#47	Rosin amine petronate	50.0
	Toluene	50.0
#48	Sarkosyl S	25.0
	Toluene	50.0
	n-Butanol	25.0
#49	Glycomul L	68.8
	n-Butanol	31.2
#50	Tetra ethyl pyrophosphate	95.6
	n-Butanol	4.4
#51	Tetraisopropyl titanate	5.0
	Sovasol #1 (dried)	95.0
#52	Tricresyl phosphate	80.0
	n-Butanol	20.0
#53	Triethanolamine phosphate	56.1
	n-Butanol	21.9
	Methanol	22.0
#54	Triethyl phosphate	91.0
	n-Butanol	9.0

Formula Number	Composition (active ingredient underlined)	Per Cent by Weight
#55	Ultrapole S	78.9
	Toluene	15.8
	n-Butanol	5.3
#56	Zinc naphthenate (8%)	100.0

TABLE II

RESULTS OF TESTS GROUPED BY

CLASSES OF MATERIALS

A. ORGANIC ACIDS

Material	Metal Finish (microns)	Film Thickness (mils)	Rust %
Emersol 150 (Stearic acid)	12	0.2	10.0
Hyfac 430 (Fish fatty acids)	1	<0.1	6.0
Emtall 662 (Rosin acids)	1	0.1	20.0
Corrosion Inhibitor NPA (Nonylphenoxy acetic acid)	1	0.3	1.0
Naphthenic acids	1	0.2	3.0
Alox 2018 (mixture of organic acids, oxy acids, and ester derived from oxidation of petroleum hydrocarbons)	1	0.2	2.0
Alox 2028 (similar to Alox 2018)	12	0.2	1.0
	4	<0.1	4.5
	1	0.3-0.4	0.0
Dodecyl benzene sulfonic acid	4	0.2	100.0
Deriphat 170C (N-lauryl B-amino propionic acid)	1	0.2	5.0

B. ESTERS

Material	Metal Finish (microns)	Film Thickness (mils)	Rust %
Metholene 221B (Methyl stearate)	1	0.1	10.0
Glyceryl mono ricinoleate	1	0.1	5.0
Glycomul L (Sorbitan monolaurate)	1	<0.1	20.0
Atpet 200 (Sorbitan partral fatty esters)	12	0.2	70.0
	12	0.2	50.0
Epoxol 9-5 (Epoxidized fatty esters) ●	1	0.2	10.0
Myverol type 18-98 (Mono glyceride of oleic and linoleic acids)	12	0.2	100.0
Alox 2018 (mixture of organic acids, oxy acids, and esters derived from oxidation of petroleum hydrocarbons)	1	0.2	2.0
Alox 2028 (see Alox 2018)	12	0.2	1.0
	4	<0.1	4.5
	1	0.3-0.4	0.0
Dilauryl thiodipropionate	1	<0.1	10.0
Methyl p-toluene sulfonate	1	0.1	30.0
Tricresyl phosphate	1	<0.1	20.0
Triethyl phosphate	1	<0.1	80.0

	Metal Finish (microns)	Film Thickness (mils)	Rust %
C. NITROGEN CONTAINING MATERIALS			
Material			
Alamine 26 D (distilled primary tallow amine)	4	<0. 1	3. 0
Ultrapole S (amine condensate)	1	0. 1	30. 0
Deriphat 170 C (N-lauryl B-amino propionic acid	1	0. 2	5. 0
Rosin amine petronate	4	0. 1	100. 0
Alamac 26D (primary hydrogenated tallow amine acetate)	1	<0. 1	40. 0
Duomeen TDO (tallow 1, 3-propylene diamine dioleate)	12	0. 2	30. 0
Triethanol amine phosphate	1	0. 1	3. 0
Nacconol 60s (triethanol amine salt of dodecyl benzene sulfonic acid)	1	---	100. 0
Ammonium petronate	4	0. 3	10. 0
Ammonium stearate	1	<0. 1	3. 0
Sarcosyl S (stearoyl sarcosine)	4	<0. 1	20. 0
Alkaterge T (substituted oxazoline)	12	0. 2	90. 0
D. ORGANIC PHOSPHATES			
Material			
Tetraethyl pyrophosphate	1	---	100. 0
Tricresyl phosphate	1	<0. 1	20. 0
Triethanol amine phosphate	1	0. 1	3. 0
Triethyl phosphate	1	<0. 1	80. 0
E. SULFUR CONTAINING MATERIALS			
Material			
Dodecyl benzene sulfonic acid	4	0. 2	100. 0
Nacconol 60-s (triethanol amine salt of dodecyl benzene sulfonic acid)	1	---	100. 0
Ammonium petronate	4	0. 3	10. 0
Rosin amine petronate	4	0. 1	100. 0
	12	0. 2	3. 0
Neutral barium petronate	4	<0. 1	10. 0
Methyl p-toluene sulfonate	1	0. 1	30. 0
Dilauryl thiodipropionate	1	<0. 1	10. 0

	Metal Finish (microns)	Film Thickness (mils)	Rust %
F. METAL SALTS OF ORGANIC ACIDS			
Material			
Neutral barium petronate	12	0.2	3.0
	4	<0.1	10.0
Calcium oleate	1	0.1	2.0
Zinc naphthenate	1	<0.1	0.0
G. POLYMERIC FILM-FORMING MATERIALS			
Material			
Acryloid B-72 (acrylic ester polymer)	12	0.2	5.0
	4	<0.1	10.0-50.0
	4	0.3	0.0
Epon 1007 (epi-chlorohydrin bis-phenol A epoxy resin)	4	<0.1	50.0-100.0
Emralon 320 (resin-bonded tetrafluoro - ethylene surface coating)	1	<0.1	0.0
Gelva C5-V16 (alkali-soluble vinyl acetate resin)	4	0.3	0.0
H. PROPRIETARY MATERIALS CONFORMING TO GRADE 4 COMPOUNDS OF MIL-C-16173b			
Material			
Clarco #4000	4	0.4	20.0
	4	<0.1	40.0
Cosmoline 1091	4	0.8	0.0
	4	<0.1	5.0
Gulf No-Rust D	4	1.0	0.0
	4	<0.1	0.0
Nokorode 733A	4	0.8	0.0
	4	<0.1	8.0
I. MISCELLANEOUS AND PROPRIETARY MATERIALS			
Material			
No-Ox-Id 493	12	0.2	3.0
Nopocochex RD	4	0.4-1.0	1.0-5.0
	1	0.4	1.0
Titanium dioxide	4	---	100.0

TABLE III

MATERIALS POSSESSING SIGNIFICANT PRESERVATIVE PROPERTIES
(Less than 10% rust when applied at thickness of less than 0.0005 inches)

Material	Metal Finish (microns)	Film Thickness (mils)	Rust %
Acryloid B-72	12	0.2	5
	4	0.3	0
Alamine 26D	4	<0.1	3
Alox 2018	1	0.2	2
Alox 2028	12	0.2	1
	4	<0.1	5
	4 & 1	0.3	0
Ammonium stearate	1	<0.1	3
Calcium oleate	1	0.1	2
Corrosion Inhibitor NPA	1	0.3	1
Cosmoline 1091	4	<0.1	5
Deriphat 170C	1	0.2	5
Emralon 320	1	<0.1	0
Gelva C5-V16	4	0.3	0
Glyceryl monoricinoleate	1	0.1	5
Gulf No-Rust D	4	<0.1	0
Hyfac 430	1	<0.1	6
Naphthenic acids	1	0.2	3
Neutral barium petronate	12	0.2	3
Nokorode 733A	4	<0.1	8
Nopcochex RD	4	0.4	5
	1	0.4	1
No-Ox-Id 493	12	0.2	3
Triethanolamine phosphate	1	0.1	3
Zinc naphthenate	1	<0.1	0

TABLE IV

WEAR DATA FALEX LUBRICANT TESTER

Time (minutes)	Test Pin Coated With: (Formula #12) Alox 2028 (inch-lbs) (Gear teeth) Torque-Wear	(Formula #4) Acryloid B-72 Torque-Wear	(Formula #32) Gelva C5-V16 Torque-Wear	None Torque-Wear
0	6 - 0	6 - 0	7 - 0	7 - 0
15	6 - 0	6 - 0	8 - 0	7 - 0
30	7 - 1	6 - 0	8 - 0	7 - 1
45	7 - 1	7 - 1	8 - 0	8 - 1
60	7 - 1	8 - 1	9 - 0	8 - 2
75	7 - 1	8 - 2	9 - 1	8 - 2
90	7 - 2	8 - 2	9 - 1	8 - 2
105	7 - 2	8 - 2	9 - 2	9 - 2
120	7 - 2	8 - 2	9 - 2	8 - 3
135	7 - 3	8 - 3	9 - 2	8 - 3
150	7 - 3	7 - 3	9 - 3	8 - 3
165	7 - 3	8 - 3	9 - 4	8 - 3
180	7 - 3	9 - 3	9 - 4	8 - 3

APPENDIX I

DETAILS OF CONTROLLED CYCLIC CONDENSATION HUMIDITY CABINET

The essential parts that are necessary for the controlled cyclic condensation humidity cabinet are listed below:

Code	Part	Description	Purpose
A	Needle valve	--	Shut-off valve for compressed air-line
B	Filter, sintered bronze	--	Remove water and oil from air
C	Pressure regulator	--	Reduce air-line pressure to suitable level
D	Filter, Fiberglas	Sealed 5-gallon drum filled with Fiberglas	Remove any oil, water, or dirt from air not removed by "B"
E	Flowrater	Flowmeter tube and valve	Control rate of air-flow through moisture chamber
F	Moisture chamber	5-gallon sealed drum with heated distilled water bath	To saturate air with water vapor
G	Heat exchanger	Copper coil in 5-gallon heated oil bath	Preheat and "dry" water vapor-saturated air
H	Air distribution manifold	Perforated copper tubing shaped in conjoined square and circle	To distribute air in humidity cabinet
I	Cabinet	Cubic, copper-sheeted box with 4 windowed doors and see-through Plexiglas cover	Contain test specimens, test atmosphere, and auxiliary equipment necessary for maintaining desired conditions.

Code	Part	Description	Purpose
J	Compressor	1/2 h.p. hermetically sealed refrigeration compressor	To enable cooling of specimens
K	Programmer	24-hour electric clock with on-off plugs	To control on-off operation of compressor
L	Moisture filter	Silica gel cartridge	To remove any vapor from refrigerant to prevent valve freeze-ups, etc.
M	Needle valves (2)	--	To isolate refrigeration manifold and return line from compressor
N	Expansion valve	--	Allow expansion of liquid refrigerant to gas
O	Manifold	1/2" copper tubing joined through T-valves	For distribution of coolant to water-jacket trays
P	Bleeder	Needle valve at end of manifold	To bleed manifold of excess refrigerant, oil; to draw vacuum, etc.
Q	Specimen trays	Stainless steel water jackets with 3/8" copper coil, with cut-out holes for mounting specimens.	To mount test specimens
R	Return line	Same as manifold	To return refrigerant gas for recirculation

AUXILIARY PARTS

Code	Part	Description	Purpose
D_1	Relief tube	3/8" open-ended, vertical, S-shaped copper tube filled with 10" column of mercury	Prevent air pressure buildup in system

Code	Part	Description	Purpose
E_1	Flowmeter	--	See "E"
E_2	Needle valve	--	See "E"
F_1	Water tank	5-gallon distilled water bottle with necessary tubing	To supply water to moisture chamber
F_2	Float valve	Small valve with float-actuated handle connected to F_1 with 3/8" copper tubing	To control water level in moisture chamber
F_3	Distributor	3/8" copper tubing with closed end and perforated sides beneath water level, leading from E_2	To bubble air through moisture chamber
F_4	Heating system	(a) Immersion heater (b) Relay (c) Thermostat (d) Thermometer (e) Pilot light	To control temperature of water in moisture chamber
G_1	Copper coil	50-foot coil of 1/2" copper tubing	Conduct air through heat exchanger
G_2	Heating system	Same as F_4	Control temperature in heat exchanger
I_1	Heating	Same as F_4 and G_2 except two heaters are used	To heat air in cabinet to desired temperature
I_2	Doors	20" x 20" hinged, Plexiglas, windowed (18" x 18") catch-locked doors	Observation; access to cabinet
I_3	Cover	30" x 30" double-layer Plexiglas lid	To enclose test while permitting observation of test

Code	Part	Description	Purpose
J1	Pressure gauge	--	For use with compressor
L_1	Sight glass	--	For use with moisture filter; indicates amount of moisture present and amount of refrigerant in system
Q_1	Connecting tubing	3/8" flare nut-fitted copper tubing	To connect water jackets with manifold (M) and return line (R)
Q_2	Rubber gaskets (2 each)	--	To prevent metal to metal contact between specimen and tray.
Q_3	Hold-down plate	Cut-out aluminum plate fitted over specimen tray	To seal specimens against water in water-jacket

For purposes of convenience, the component parts of the controlled cyclic condensation humidity cabinet are grouped under one of three classifications. These classifications are:

(1) Air Distribution and Moisture Control System
(2) Cabinet
(3) Refrigeration and Specimen Holder Systems

Included in the Air Distribution and Moisture Control System are the following component parts: (These parts have been lettered in accordance with the sequence of operations.)

A - Needle valve	E - Flowrater
B - Sintered bronze filter	F - Moisture chamber
C - Pressure regulator	G - Heat exchanger
D - Fiberglas filter	H - Air distribution manifold

The Refrigeration and Specimen Mounting System is composed of the following parts: (These, as with the air distribution and humidity control system, are lettered in accordance with the sequence of operation)

J - Compressor	O - Manifold
K - Programmer	P - Bleeder
L - Moisture filter	Q - Specimens trays
M - Needle valves	R - Return line
N - Expansion valve	

The cabinet (I) houses the air distribution manifold (H) of the Air Distribution & Humidity Control System and the expansion valve (N), manifold (O), bleeder (P), specimen trays (Q), and return line (R) of the Refrigeration and Specimen Mounting System.

In general, the systems and component parts of our Controlled Cyclic Condensation Humidity Cabinet correspond rather closely in function and construction with those in the equipment constructed by Minuti at Naval Air Material Center Laboratories, Philadelphia, Pennsylvania.

In the Air Distribution and Humidity Control System, the upper section of the air distribution manifold (H) has been enlarged in order to accommodate the different design of the specimen trays (Q). The cabinet is constructed as the original cabinet. However, in the Refrigeration & Specimen-Mounting System, several changes have been made. The specimen trays (Q) have been redesigned to accommodate ten 2" x 4" test specimens each, whereas the original specimen trays could accommodate only one 6" x 6" test specimen. In the original equipment, nine specimen trays (6" x 6" specimens in rows of 3 each) were contained in the cabinet, while in our equipment, there are 5 specimen trays (ten 2" x 4" specimens) inside the cabinet. These new trays are supported by two aluminum bars anchored to the side walls of the cabinet. The manifold and return line construction was necessarily modified to conform to the new specimen tray arrangement. T-valves are used to connect specimen tray refrigeration coils with the manifold and return lines because they offer a greater variety of control of the refrigeration cycle than was possible in the previous arrangement. The bleeder valve (P) and needle valves (M) were included in the system so that the specimen trays could be more easily isolated from the compressor when repairs are necessary, or samples are changed.

APPENDIX II

SOURCES OF ACTIVE INGREDIENTS

Supplier	Reference Formula No.
Acheson Colloids Company Port Huron, Michigan	27
Alox Corporation P.O. 566 Niagara Falls, N.Y.	10, 11, 12
Armour Industrial Chemical Co. Division of Armour & Company 110 N. Wacker Drive Chicago 6, Illinois	25
Atlas Chemical Industries, Inc. Chemicals Division Wilmington 99, Del.	15
Commercial Solvents Corporation 260 Madison Avenue New York 16, N. Y.	9
Clarkson Laboratories Inc. 1450 Ferry Avenue Camden, N. J.	17, 18
Dearborn Chemical Company Merchandise Mart Plaza Chicago 54, Illinois	45
Distillation Products Industries Division of Eastman-Kodak Co. Rochester, N. Y.	38, 39
E. I. Du Pont de Nemours, Inc. Wilmington 98, Delaware	22, 51
Eastman Chemical Products, Inc. Subsidy of Eastman-Kodak Co. Kingsport, Tenn.	52, 54
Emery Industries Inc. 2505 Carew Tower Cinncinnatti 2, Ohio	26, 28, 36, 37

Supplier	Reference Formula No.
Fisher Scientific Company 203 Fisher Bldg. Pittsburgh 19, Pa.	14, 16
Geigy Chemical Corp. Saw Mill River Rd. Ardsley, N. Y.	19, 48
General Mills, Inc. Chemical Division Kankakee, Illinois	7, 8, 23
Glyco Products Co., Inc. 417 Fifth Avenue New York 16, N. Y.	33, 49
Gulf Oil Corp. 360 Lexington Ave. New York 17, N. Y.	34, 35
Harshaw Chemicals Company 1945 East 97th Street Cleveland 6, Ohio.	56
E. F. Houghton & Company 303 W. Lehigh Avenue Philadelphia 33, Pa.	20, 21
Lion Oil Company Division of Monsanto Chemical Co. El Dorado, Arkansas	42, 43
Matheson, Coleman & Bell, Inc. East Rutherford, N. J.	40
A. R. Maas Chemical Company Division of Victor Chemical Works South Gate, California	53
Monsanto Chemical Company 800 N. Lindbergh Blvd. St. Louis 66, Mo.	50
National Amiline Div. Allied Chemical & Dye Corp. 40 Rector Street New York 6, N. Y.	46

Supplier	Reference Formula No.
Nopco Chemical Company 60 Park Place Newark, N. J.	44
Rohm & Haas Company Washington Square Philadelphia 5, Pa.	1, 2a, 3, 4, 5, 6
Shawinigan Resins Corp. Springfield 1, Mass.	32
Shell Chemical Company Industrial Chemicals Div. 50 West 50th Street New York 20, N. Y.	29
Sonneborn Chemical & Refining Corp. 300 Park Avenue S. New York 10, N. Y.	2b, 13, 41, 47
Swift & Company Industrial Chemicals Div. 115 W. Jackson Blvd. Chicago 4, Illinois	30, 31
Ultra Chemical Works Div. of Witco Chemical Co., Inc. 122 E. 42nd Street New York 18, N. Y.	24, 55

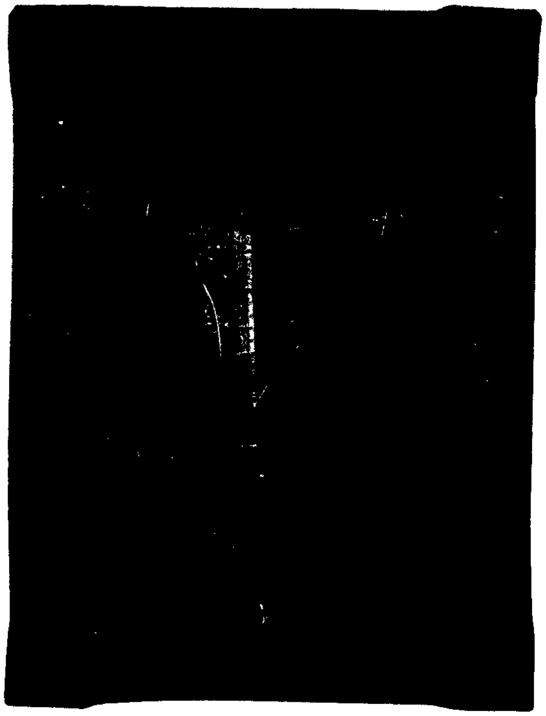
SPECIMEN MOUNTING TRAY AND REFRIGERANT MANIFOLD

SPECIMEN MOUNTING TRAY

REFRIGERANT MANIFOLD

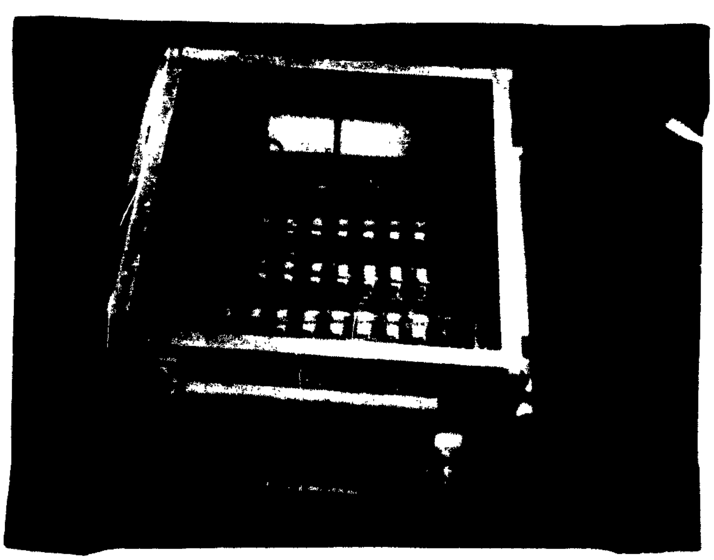
PLATE #1

CONSTRUCTION OF CONTROLLED CYCLIC CONDENSATION HUMIDITY CABINET



1. INSULATED TEST CABINET SHELL

2. CABINET WITH DOORS, REFRIGERANT AND AIR DISTRIBUTION MANIFOLDS INSTALLED



3. CABINET WITH DOORS, REFRIGERANT AND AIR DISTRIBUTION MANIFOLDS, AND SPECIMEN MOUNTING TRAYS INSTALLED

PLATE #2

CONTROLLED CYCLIC CONDENSATION HUMIDITY CABINET

Top View of Moisture Chamber (F)

Scale: 1/2" = 1"

F_1	Water feed tank (not shown)		F_{4c}	Thermostat	
F_2	Float valve		F_{4d}	Thermometer	
F_3	Air Distributor		F_{4e}	Pilot light	(not shown)
F_{4a}	Immersion heater		E	Flowrater	(not shown)
F_{4b}	Relay	(not shown)	G	Heat exchanger	(not shown)

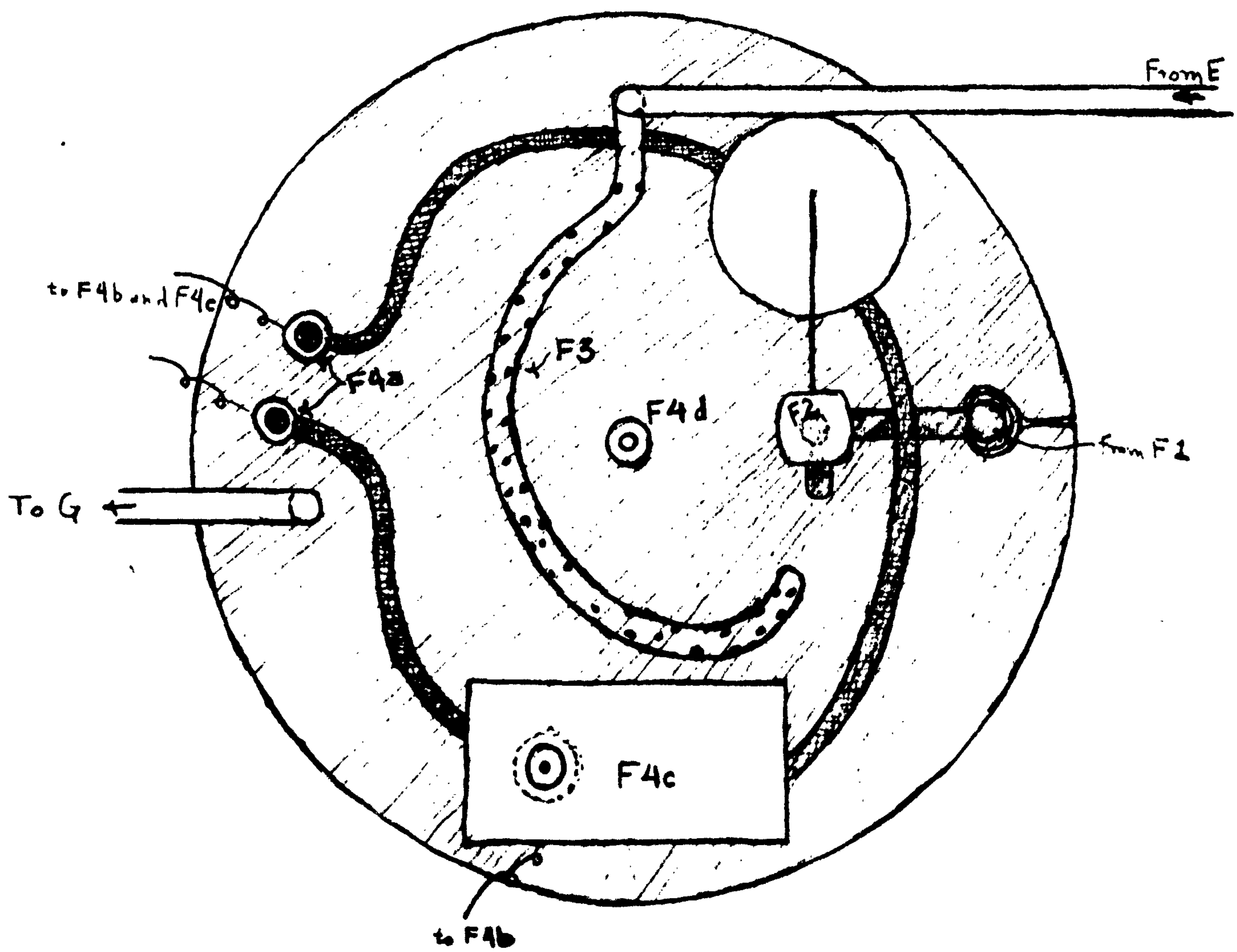


PLATE #3

CONTROLLED CYCLIC CONDENSATION HUMIDITY CABINET

Side view of Moisture Chamber (F) | **Scale: 1/2" = 1"**

F-1	Water feed tank (not shown)	F-4c	Thermostat	
F-2	Float valve	F-4d	Thermometer	
F-3	Air distributor	F-4e	Pilot light	(not shown)
F-4a	Immersion heater	E	Flowrater	(not shown)
F-4b	Relay (not shown)	G	Heat exchanger	(not shown)

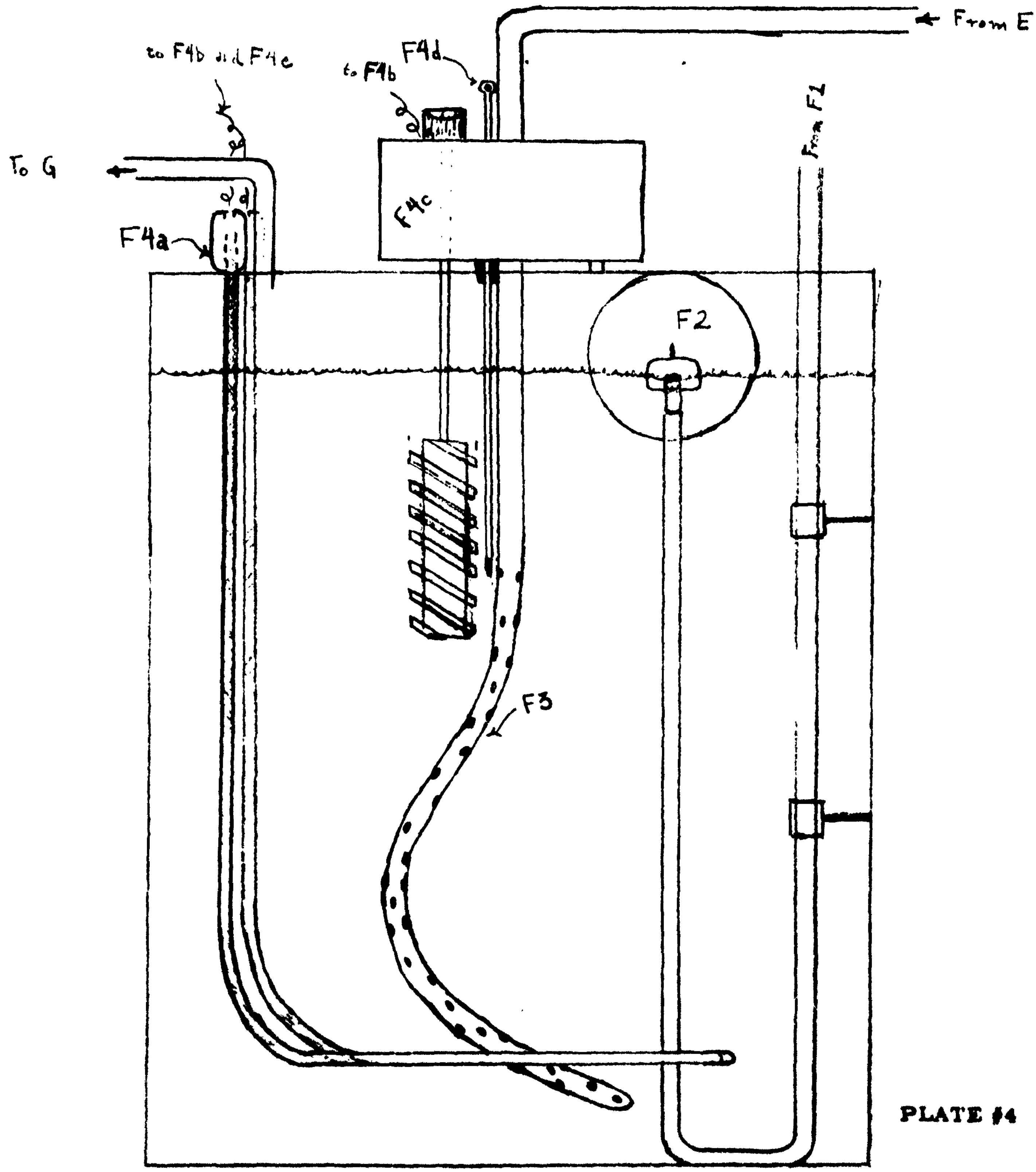
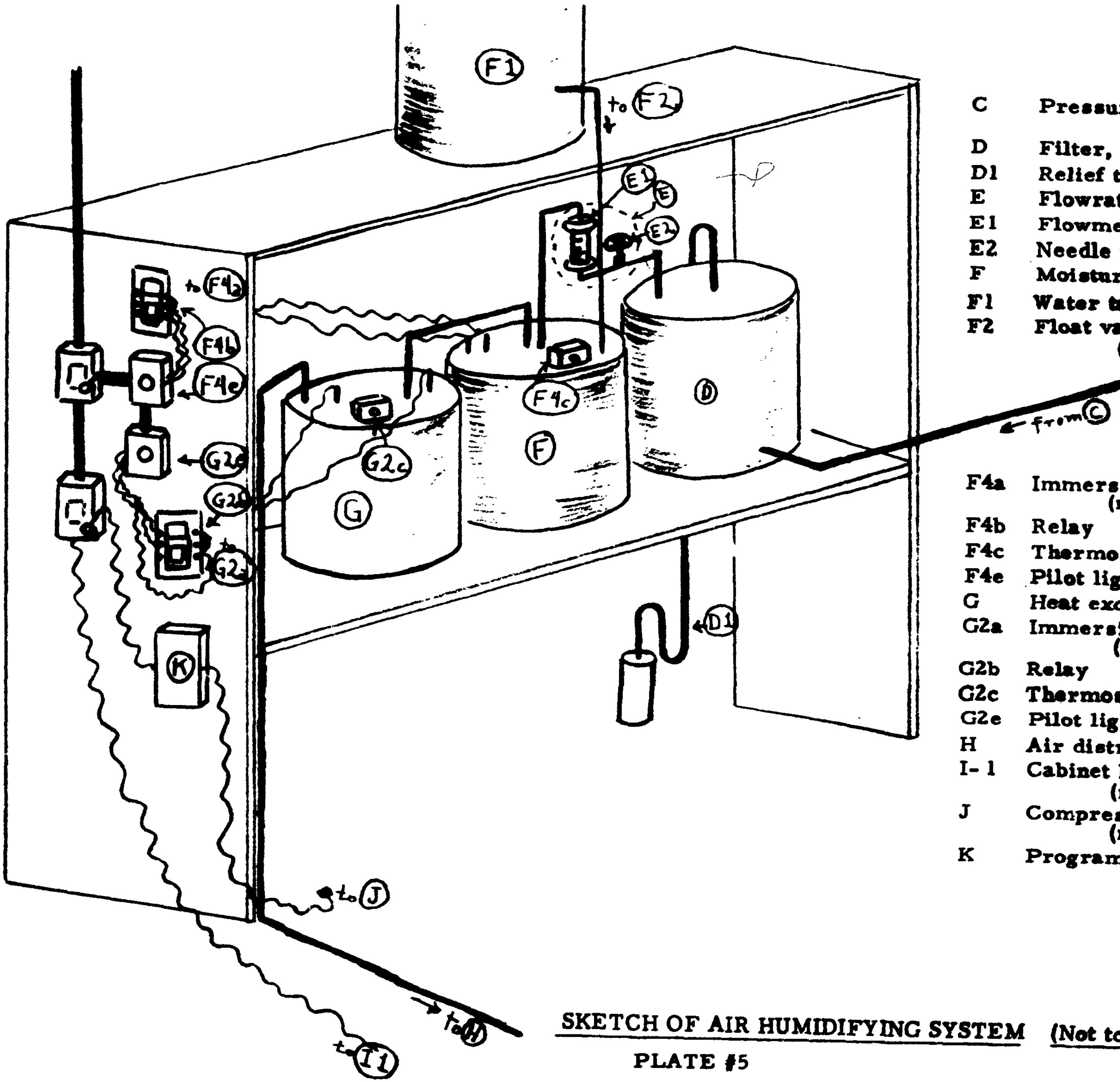


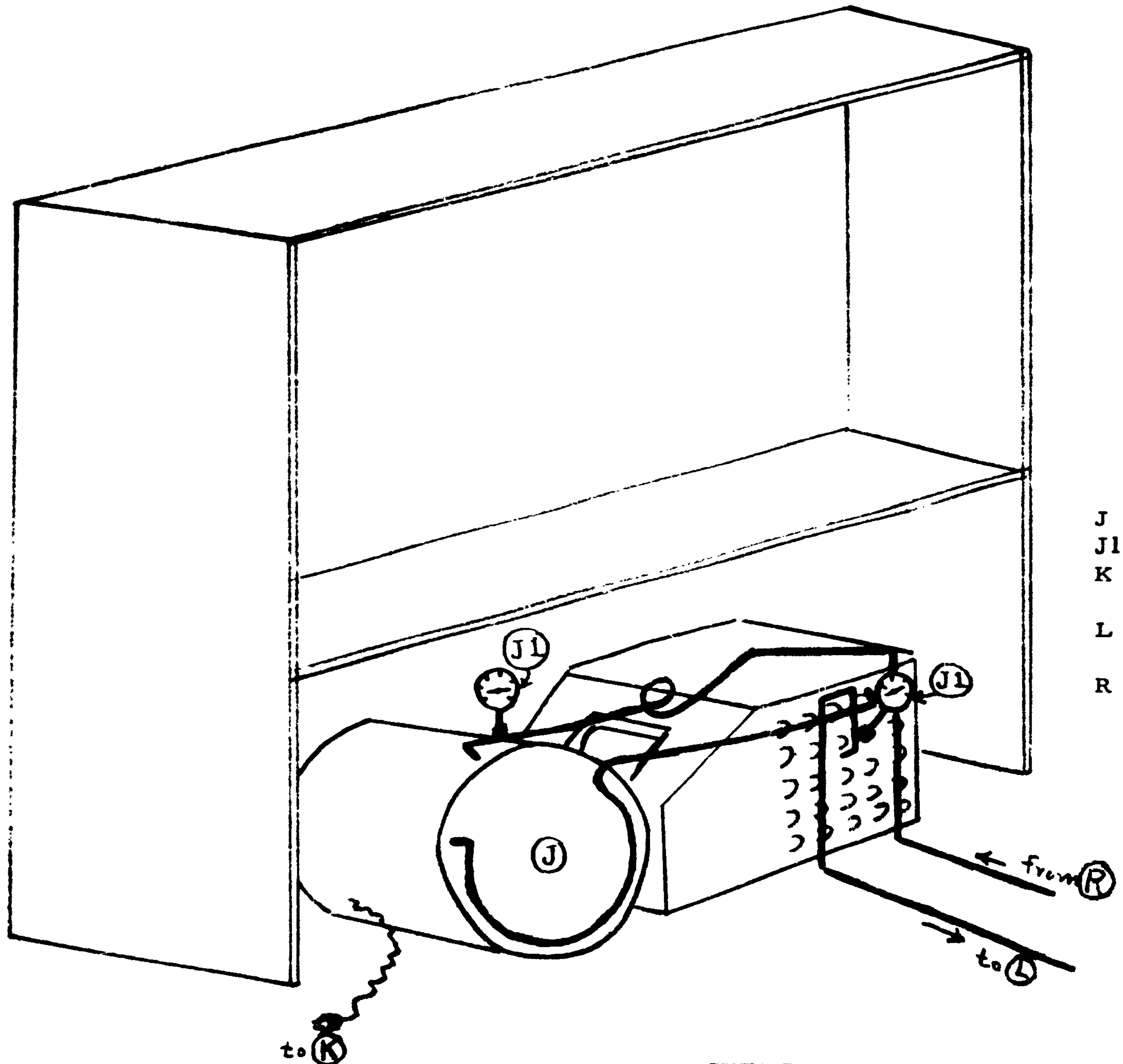
PLATE #4



C	Pressure regulator (not shown)
D	Filter, Fiberglas
D1	Relief tube
E	Flowrater
E1	Flowmeter
E2	Needle valve
F	Moisture chamber
F1	Water tank
F2	Float valve (not shown)
F4a	Immersion heater (not shown)
F4b	Relay
F4c	Thermostat
F4e	Pilot light
G	Heat exchanger
G2a	Immersion heater (not shown)
G2b	Relay
G2c	Thermostat
G2e	Pilot light
H	Air distribution
I-1	Cabinet heating system (not shown)
J	Compressor (not shown)
K	Programmer

SKETCH OF AIR HUMIDIFYING SYSTEM (Not to Scale)

PLATE #5

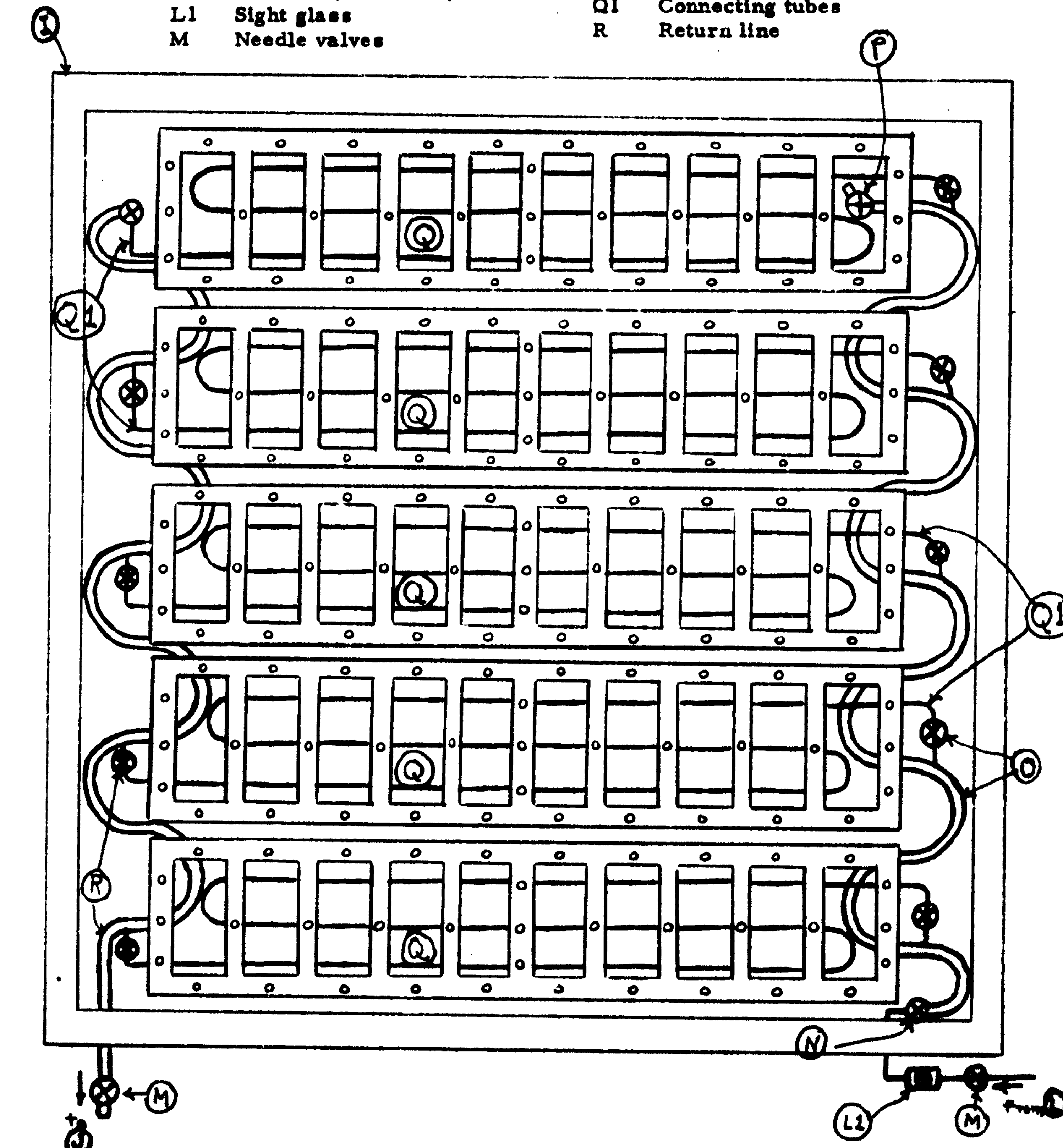


J	Compressor
J1	Pressure gauges
K	Programmer (not shown)
L	Moisture filter (not shown)
R	Return line (not shown)

SKETCH OF COMPRESSOR (Not to Scale)

PLATE #6

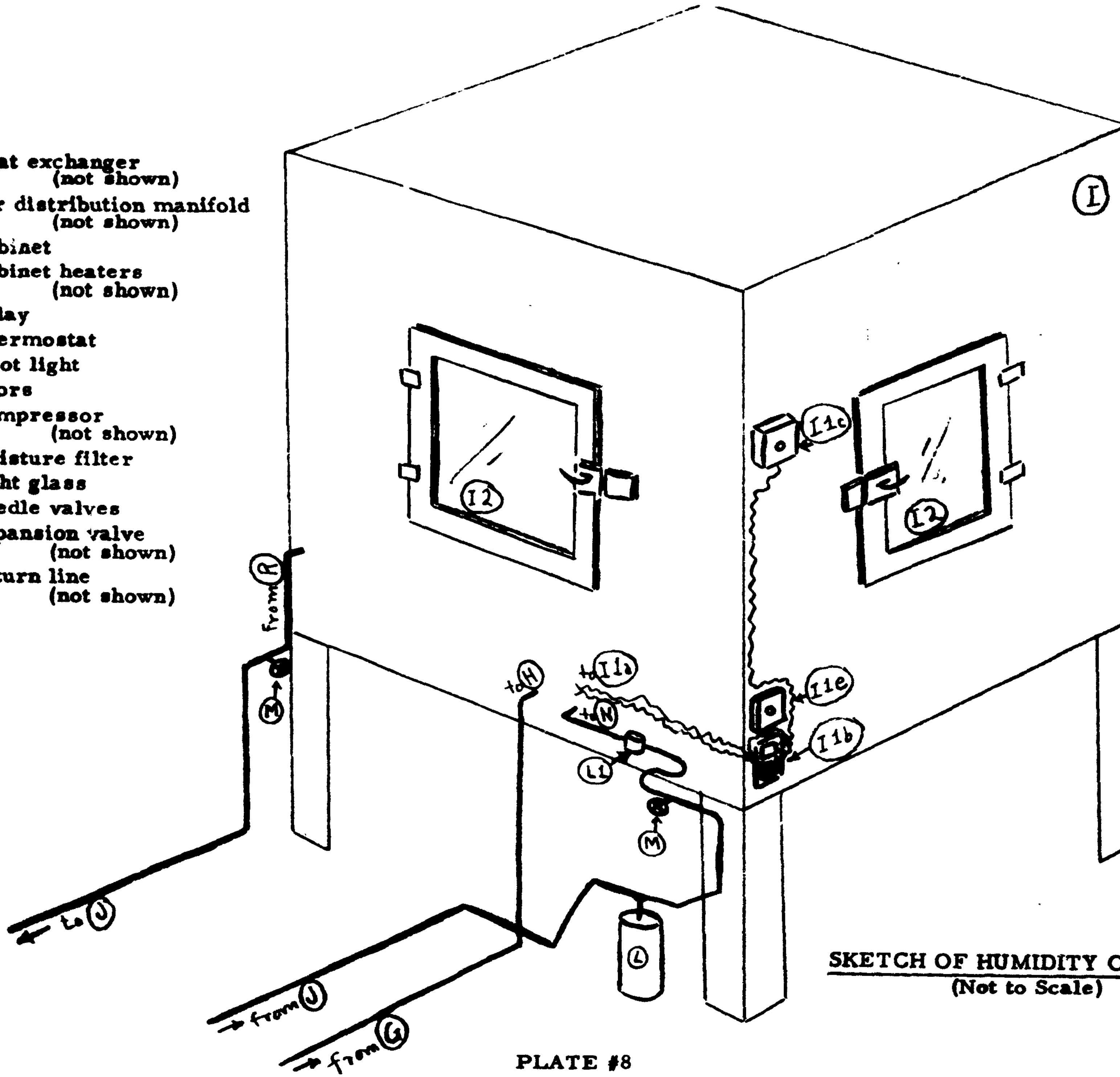
I	Humidity cabinet	N	Expansion valve
J	Compressor (not shown)	O	Manifold and T-valves
L	Moisture filler (not shown)	P	Bleeder
L1	Sight glass	Q	Specimen trays
M	Needle valves	Q1	Connecting tubes
		R	Return line



SKETCH (Top View) OF TEST PANEL MOUNTING (Not to Scale)

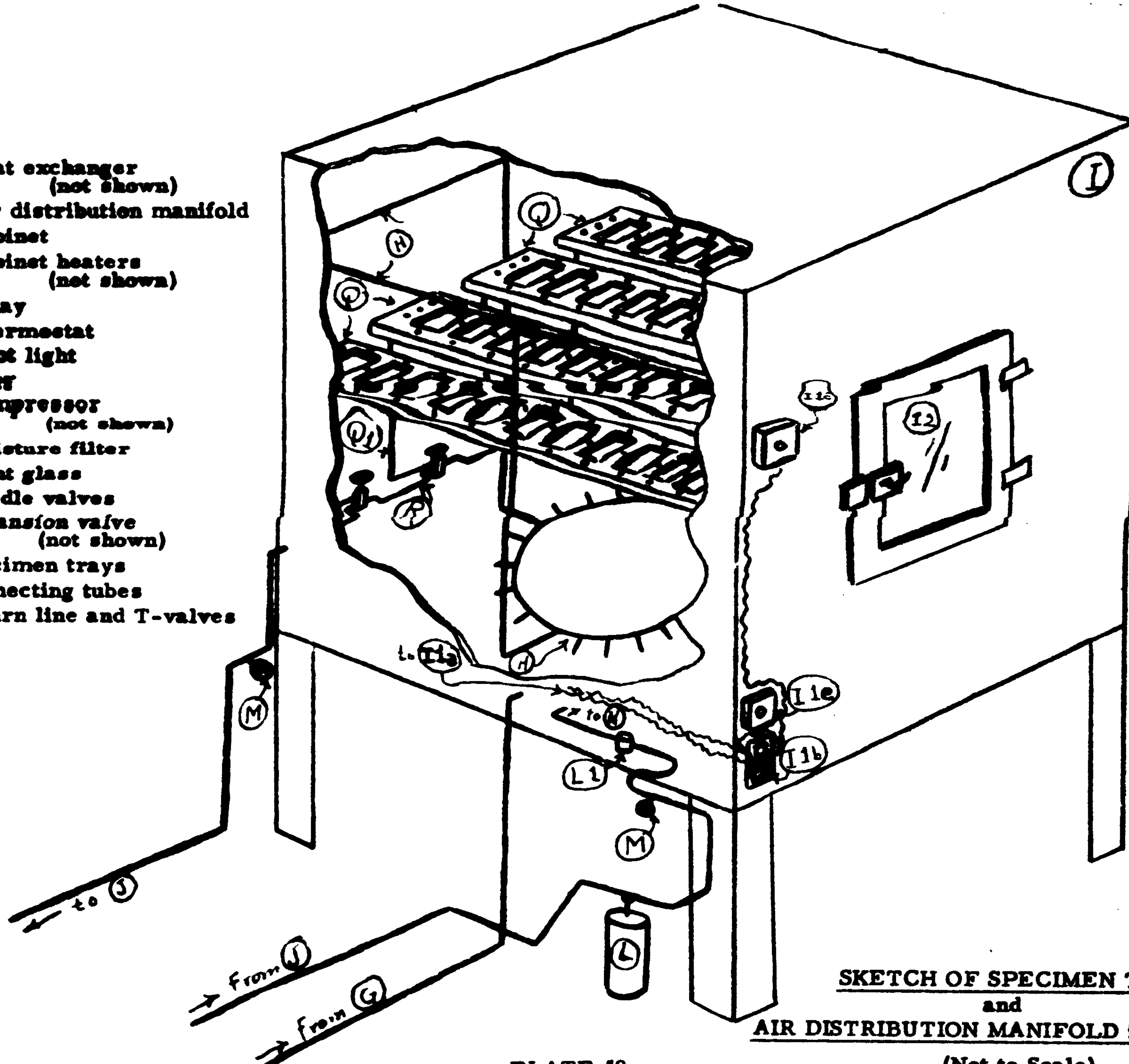
PLATE #7

G	Heat exchanger (not shown)	
H	Air distribution manifold (not shown)	
I	Cabinet	
I-1a	Cabinet heaters (not shown)	
I-1b	Relay	
I-1c	Thermostat	
I-1e	Pilot light	
I-2	Doors	
J	Compressor (not shown)	
L	Moisture filter	
L1	Sight glass	
M	Needle valves	
N	Expansion valve (not shown)	
R	Return line (not shown)	



SKETCH OF HUMIDITY CABINET
(Not to Scale)

PLATE #8



G	Heat exchanger (not shown)
H	Air distribution manifold
I	Cabinet
I-1a	Cabinet heaters (not shown)
I-1b	Relay
I-1c	Thermostat
I-1e	Pilot light
I-2	Door
J	Compressor (not shown)
L	Moisture filter
L1	Sight glass
M	Needle valves
N	*Expansion* valve (not shown)
Q	Specimen trays
Q1	Connecting tubes
R	Return line and T-valves

PLATE #9

SKETCH OF SPECIMEN TRAYS
and
AIR DISTRIBUTION MANIFOLD in CABINET
(Not to Scale)